



Standard Procedure For Collection of Coating and Ink Samples For Analysis By Reference Methods 24 and 24A



**STANDARD PROCEDURE FOR
COLLECTION OF COATING AND INK SAMPLES FOR
ANALYSIS BY REFERENCE METHODS 24 AND 24A**

**U.S. ENVIRONMENTAL PROTECTION AGENCY
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Office of Air Quality Planning and Standards
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Washington, DC 20460**

September 1991

DISCLAIMER

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SECTION 1

INTRODUCTION

This standard operating procedure (SOP) document is prepared with the intent of providing simple step-by-step instructions, covering all aspects of sampling coatings and inks, for use by EPA, State, and local regulatory agencies nationwide. The instructions are presented in general terms as much as possible while providing sufficient details for actual field sampling. The procedure should be used in conjunction with existing health and safety programs and in accordance with existing EPA or other agency training guidelines.

Standard procedures are presented for sampling and handling of coatings and inks that require analysis by EPA Reference Methods 24 or 24A (RM 24/24A) as found in 40 CFR 60, Appendix A. This SOP has been expanded from an original version obtained from EPA Region II. For the purpose of developing consistency among agency inspectors nationwide in terms of sampling conducted, it delineates the activities deemed proper and necessary to ensure that the sample taken is representative of the coating or ink as applied. If the activities are conducted as described, questions concerning the credibility of sampling performed can be avoided.

Agency inspectors are generally responsible for a) planning for sampling (*i.e.*, deciding on the date, process, and location of sampling), b) ensuring that the samples are drawn properly, and c) handling and transporting samples to the laboratory responsible for analysis. The inspector rarely draws the sample. On-site the inspector typically requests the facility representative to assign a facility employee to draw the sample under the agency supervision, while providing the necessary equipment and guidance for sampling.

The use of common sense and care are required in procuring and submitting representative samples of industrial surface coatings and printing inks for analysis. Several EPA offices as well as State and local agencies have developed procedures for their staff, covering various aspects of sampling (*i.e.*, sampling techniques, containers, as well as storage and transfer of samples.) ASTM also has published several specific and detailed sampling procedures.^{1,2,3,4}

The Standard Operating Procedure (SOP) presented in this document covers all types of industrial coating and printing ink operations, regardless of the method of application of coating or ink (*i.e.*, dip, spray, roll, flow, electrostatic, or electro-deposition). The only exceptions are the source categories such as consumer, architectural, or aerosol coatings, for which little compliance sampling experience is available. For these categories, the local process conditions, such as the coating storage and application methods, may dictate some minor changes in the way the sample is collected.

The sampling, handling, labeling, chain of custody, and quality assurance/ quality control conditions and procedures are addressed in the following sections. The equipment, supply material, data recording forms, and labels that must be either carried or readily available to the inspector in the field are listed below. However, the actual use of equipment will depend upon the local process conditions existing at the source sampled.

Sampling Supplies:

- Dual seal tin sample containers, one cup (8 fl.oz.), 1 pint (16 fl.oz.), 1 liter (equivalent to 1 quart or 32 fl.oz.) or special plastic sample containers for corrosive coatings
- Waterproof/solvent-proof marking pen
- Small scraper or knife/spatula
- Clean rag, paper towels
- Cooler/ice
- Long handled tongs
- Tubing
- Mixing/stirring paddles

Personal Safety Equipment:

- Eye protection
- Respiratory protection
- Hearing protection
- Steel toe shoes and gloves
- Hard hat
- Anti-sparking equipment (clamp-ended grounding cables)

Shipping Supplies:

- Packing and/or shipping box(es)

- Packing material: bubble paper, newspaper
- Shipping labels/forms
- Strapping tape

Data Recording Forms and Labels:

Listed below are standard forms and labels which will be required to properly record and identify samples. The inspector should plan to have an ample supply on hand at the source as deemed necessary.

- Coating Data Sheets⁵ - (see Appendix A)
- Chain of Custody (C of C) strip seal (see Appendix B)
- Sample can label (see Appendix C)
- Chain of Custody (C of C) Record (see Appendix D)

SECTION 2

PROCUREMENT OF SAMPLES

The recommended step-by-step activities for proper sampling are presented below:

1. Confirm sample analysis arrangements (with the laboratory assigned to perform the analysis) prior to taking samples, particularly if they are non-routine.
2. Identify coatings or inks and the processes from which they are to be sampled:
 - a. Identify the location in the operation where samples can best be obtained. The sample should be taken at the point of application of coating or ink, or as close to that point as possible, in order for the sample to be representative of the coating material "as applied" to the web or substrate.
 - b. Multi-component coatings that harden upon mixing and application to the substrate must be sampled differently since a representative "as applied" sample cannot be obtained in the field. Examples of these coatings are the two and three part catalyzed polyurethane coatings. Each component of these coatings must be sampled separately and submitted for laboratory analysis as a multi-part sample. The component mix ratio must be obtained from the facility at the time of sampling and submitted to the analytical laboratory. This will enable the laboratory to mix the components using the same proportions as in the actual coating operation prior to the analysis.
3. Make sure the coating is thoroughly mixed before sampling. During operation of the coating application equipment, the coatings may be shaken mechanically or stirred with various agitators or circulation systems. Lacquers and other coatings containing highly volatile solvents should be agitated in closed containers to avoid evaporation. Water-thinned coatings tend to incorporate air bubbles if stirred too vigorously, so they should be stirred slowly. The risk of stratification or separation of components into a non-homogeneous mixture depends on the type of coating and is also directly proportional to the size of the reservoir being sampled. To ensure that a representative sample is obtained, it is essential that it be taken during steady process operation and at the point of application to the web or substrate (or as close to it as possible).
4. Most New Source Performance Standards (NSPS) regulations cited in 40 CFR 60 for surface coating operations require the use of at least a one liter (approximately 32 fl.oz.) sample container.

For sampling of all sources other than NSPS sources, use a tin dual seal 8 fl.oz. sampling container. A 16 fl. oz. container may be used, but it will result in excessive material for later disposal. Small 4 fl. oz. sample containers may be used for the catalyst that will be added to multi-component formulations in the laboratory since only small proportions are normally used.

Special plastic containers or glass sample bottles have been used for corrosive substances such as acid etch primers and paint catalysts such as MEK peroxide.⁶ If a plastic container is used it must be impermeable to VOC diffusion through the walls of the plastic container.¹ Sample containers, caps, and inner seal liners must be inert to the chemically reactive compounds in the sample and must therefore be selected on a special case-by case basis by the agency affected.

5. Request a copy of the blender's worksheet to obtain data on the exact coating being sampled. Also collect manufacturer's formulation information from product data sheets. The ink, coating, and solvent data should be available on-site in the form of Material Safety Data Sheets (MSDS). The MSDS forms contain recommendations for safe handling of materials as well as physical and chemical properties data. Request data on the coating cure time and conditions if not provided in product data sheets and MSDS forms. Determine if exempt solvents are present or if any special handling or safety precautions will be required.
6. Bring a blank Coating Data Sheet form⁵ (see Appendix A) for reference purposes to ensure that sufficient information is gathered from plant documents to fill out or calculate the necessary input data.
7. Only one sample is required for each coating to be characterized. The sample can be used in the analytical laboratory for a number of repeat analyses as required. One field replicate sample should be taken for every 10 samples collected. A minimum of one field replicate sample should be taken for each facility visit, even if less than 10 samples are taken. This provides a means to check the accuracy of the methods used. If a company requests a set of samples for its own analysis, an entirely separate set of samples should be taken concurrently using new sample containers. A fresh new sample should be taken if a repeat or follow-up sample is required for any reason.
8. When coating samples are procured on-site, the inspectors and other personnel should protect themselves from exposure to potentially hazardous chemicals as discussed below:
 - a. Wear proper personal protecting equipment. The MSDS may indicate the manufacturer recommended personal protective equipment (PPE) for use during handling of samples. The MSDS forms for the coating

materials, including dilution solvents, should be readily available at the point of use in the facility.

- b. If a site safety plan is in effect, adhere to all of the provisions which relate to the coating operations being sampled. These requirements may include, but are not limited to: respiratory protective devices (e.g., air purifying respirators, air-supplied respirators), protective clothing (e.g., gloves, apron), eye protection (e.g., safety goggles, face shield), hearing protection (e.g., ear plugs or muffs), use of non-sparking tools and equipment, and other requirements as deemed necessary by the on-site safety personnel.
 - c. If no safety plans, MSDS, or on-site safety personnel are available, then the manufacturer of the coating materials and/or the regional OSHA office may be contacted for guidance on safety practices and the types of PPE to be utilized during sampling.
 - d. At a minimum, the applicable federal and state safety and health laws such as those found in Title 29 of the Code of Federal Regulations, Part 1910⁷, shall be complied with by all inspection personnel supervising the sample procurement.
9. Have a sample of the selected coating or ink drawn by the facility's designated person. A clean rag or paper towels should be kept handy, since the filling of the sample container may result in some spillage.
- a. Inspect the sample container to ensure that the inside and outside are clean and dry. Then hand it to the facility operator assigned to draw the sample.
 - b. Have the sample container connected to an electrical ground using grounding clips. This is particularly important when sampling from coating operations using electrostatic or electro-deposition technologies.
 - c. Have the facility operator assigned to the task draw samples by filling each sample container one-at-a-time. Work as fast as possible to avoid loss of VOC from the sample. Depending upon the location in the process from where the sample is taken, the steps presented below must be followed in drawing samples.

Usually, a sample is taken from 1) a spray nozzle or other applicator; 2) a coating bath/agitated reservoir holding coating ready for application; or 3) a bleed valve, hose, tank, or other location upstream of the point of application. Usually it is best to take the sample at the application point, i.e., a spray gun or nozzle. When sampling at locations other than the

spray gun, one must make sure that the coating or ink is not thinned beyond the sample point.

(1) When sampling from a spray gun or other application device:

- Shut off the compressed air or atomizing fluid pressure and tilt the nozzle to about a 60° angle.
- Tilt the sample container to the same angle, insert the nozzle or application device into the sample container, and begin to fill the sample container using the liquid feed pressure to provide flow, gradually tilting the sample container upright as it fills.
- Slowly fill the container to overflowing to ensure that a representative sample is obtained and to avoid any loss of VOC due to volatilization to the headspace. Do not insert the applicator tip into the coating. This may contaminate the sample or create bubbles leading to VOC loss.
- Obtaining a representative sample from high pressure spray gun applicators or from coatings or inks that contain high vapor pressure VOC's may prove difficult.

(2) When sampling from an agitated/circulating coating bath or container holding coating ready for application:

- Wipe off the sample container or make sure it is clean before dipping it into the coating reservoir. Turn the sample container upside down and place it in the coating, approximately halfway down. (Do not take the sample from the top surface.) This can be done with a clean pair of long handled tongs to avoid contaminating the reservoir.
- Turn the sample container over and slowly bring it to the top of the coating reservoir.
- It is important to completely fill the container to avoid any loss of VOC due to volatilization to the headspace. A sample filled to the top and spilling over is acceptable. This requirement applies to the catalyst of multi-component coatings as well as to the uncatalyzed coating.

When sampling inks, a sample may be taken directly from the ink trough using a metal ladle, glass jar, or a clean

paper cup to fill the sample container. A foam cup which may dissolve upon contact with solvents should not be used.

- (3) If it is not possible to sample from the coating container or applicator, a sample may be taken from a tap, bleed valve, paint hose, drum, tank, or other location in the system which is as close to the point of application as possible and therefore will provide the best possible "as applied" coating sample. Sampling at each of these alternate points will require some judgement, since each coating line or process may have a different orientation and layout of taps, valves, hoses, and reservoirs. If additional information or guidance is needed, refer to ASTM Method D4057-88 for Standard Practice for Manual Sampling of Petroleum and Petroleum Products³ and ASTM Method E300-86 for Standard Practice for Sampling Industrial Chemicals⁴ for detailed procedures and recommendations. Basic instructions for sampling from larger reservoirs or containers are:

- Flush any tap, valve, hose, or other sample line thoroughly before sampling. Sampling from 55 gallon drums or larger tanks should be avoided because stratification or separation of components may occur under all but the most ideal mixing conditions. Hold the sample container upright or at a slight angle so that the sample tap may be inserted into the container.
- Insert the tap, valve or hose into the sample container and begin to fill it while attempting to avoid contamination of the sample by contact with external parts of the sample line.
- It is important to completely fill the container to avoid any loss of VOC due to volatilization to the headspace.

- d. Once the sample is taken, the steps presented below must be followed:

- Place the sample container on the floor or ground and insert the inner seal on the container. The most efficient way to do this is to place the seal inside the rim of container, invert a screw cap, and with the open palm of the hand press down on the screw cap; this will evenly force the inner seal into the container for a tight fit.
- Screw cap onto can.

- Wipe all residual coating material off the sample container. To avoid contamination, do not allow cleaning of the container prior to inserting the inner seal.
 - Sign and date the Chain of Custody (C of C) strip seal (see Appendix B), place it over top of the screw cap and down sides of the sample container tightly, following contours.
 - Each sample should be numbered with a unique number.
 - Completely fill out the sample container label. The label should contain the following information (An example label is given in Appendix C):
 - Name of Agency and inspector who obtained the sample.
 - Sample ID No.
 - Sample date and time.
 - Source identification and sample point (e.g., coating line and coating station identification for the collection site).
 - Sample description - color, type, solids/water content, or multi-component portion.
 - Set-up time of multi-component coatings, if applicable.
 - Plant witness (signature).
 - Analysis required - RM 24 or RM 24A.
 - Presence of exempt solvents and their identification.
 - Special handling procedures that may be required.
 - Finally, affix the completed label onto the side of the sample container over the C of C seal ends.
10. Complete the C of C form (given in Appendix D) for each sample. The inspector should retain a copy of the C of C until the original comes back from the analytical laboratory. All other copies and original should remain with the sample to be sent for analysis.

SECTION 3

CHAIN OF CUSTODY (C of C)

Chain of custody procedures are very important. They show who controlled or handled the samples. Proper documentation of each sample handler is essential to preserving the integrity of the sample and its use as evidence.

1. Each person who handles the sample must be identified on the accompanying C of C Record form. (A suitable blank C of C record form is presented in Appendix D along with a completed example.) More than one sample can be included on a C of C record if each sample is clearly identified and the analysis/handling instructions for each are clearly and unambiguously given.
2. If shipping of the sample is required, the C of C sheet should be enclosed in an envelope and should travel with the sample inside the shipping box. The analytical laboratory personnel receiving the sample are responsible for signing the original C of C and returning it to the agency.
3. If air shipping is required the Air Cargo bill of lading (or other shipping bill/receipt documents) becomes part of the C of C and should be attached to the original C of C by the person receiving the delivery.
4. Before enclosing it in the shipping box, ensure that all portions of the C of C form are filled out including specification that the RM 24 or 24A analysis is to be performed by the analytical laboratory and including identification of exempt solvents that may be present in the sample.
5. In most cases, use one C of C form for each sample.
6. The agency inspector should retain one copy of the C of C.
7. The original C of C form will be returned by the analytical laboratory receiving the sample to the designated agency representative. The C of C form should have signatures from the agency inspector, the sample custodian, the person in charge of shipping samples, and the responsible analytical laboratory representative before it is returned to the agency. When the C of C is returned to the agency, a copy of the C of C should be given to the inspector and the original kept by the person responsible for shipping or recordkeeping.

SECTION 4

QUALITY ASSURANCE/QUALITY CONTROL

Documentation

1. All required information should be completely recorded on appropriate C of C, strip seal, or label forms. Each sample recipient should check C of C, strip seal, or label forms for completeness and should not accept samples if C of C, strip seal, or label forms are incomplete. The recipient should require that any key missing information be provided to adhere with procedures intended to ensure integrity and C of C documentation of the sample.
2. Sampling activities should be documented in a logbook or recordbook in the event verification or testimony is required at a later date.
3. The C of C form should be legibly and completely filled out, including directions for which reference method analysis (*i.e.*, 24 and/or 24A) should be performed and identifying any exempt solvents that may be present.
4. MSDS or other product data sheets, blender's worksheets, coating manufacturer's data, or trade names (as well as multi-component coating formulation blend and set-up time information) should only be sent with samples to the laboratory for analysis if reference to % volatile, % H₂O, % exempt solvents, VOC lbs/gallon, specific gravity, or specific information about the origin of the sample are removed or excluded from the data. This is to ensure objective and unbiased analysis of the sample. MSDS and other qualitative information describing the sample is essential to proper and safe handling in the laboratory.
5. The person submitting the samples should indicate the presence of water, ask for % water analysis only when applicable, identify the presence of exempt solvents, and specifically request analysis for exempt solvents when present.
6. The shipping documents should be filled out correctly.

Labeling

7. C of C strip seal tape should be placed on container properly and securely.
8. The sample label should be completely and accurately filled out indicating the analysis requested, the presence of exempt solvents, or if special handling is required. It should be placed on the container so that it is readily visible.

9. When the sample contains an exempt solvent or a multi-component coating to be specially blended (mixed) by the analytical laboratory, a special tag should be completed and affixed to the sample (such as a 2½" by 4½" label tied to the sample can with a string) to notify the lab that the sample requires special handling or analysis.
10. The shipping labels should be filled out correctly.

Sample Integrity

11. The sample container should be filled to overflowing prior to placing inner seal in container. A partially filled container indicates potential VOC loss or some other problem with the sample. A repeat sample is required.
12. To avoid potential sample contamination, the outside of the sample container should be wiped clean immediately after being sealed.
13. If analytical laboratory personnel receive samples stored in inappropriate containers or if there are any other problems associated with sample integrity or documentation, they should immediately report these problems to agency personnel.

SECTION 5

HANDLING

Simple common sense should be used in handling collected samples to ensure sample integrity and custody control.

1. Maintain samples at room temperature, preferably at 70°F but within the range of 40 to 100°F. Do not store sample containers in hot areas such as a closed vehicle. Keep out of direct sunlight and keep from freezing.
2. After properly sealing the sample container, the sample should be secured in a safe place and maintained at a temperature that is compatible with the coating. The packing requirements of individual samples depend on the coating's physical and chemical properties. Coatings with relatively high vapor pressures (*i.e.*, those containing volatile solvents with boiling points below 100°F) should be packed in ice to keep them within the temperature range of 40 to 100°F. The inspector or person responsible for samples should use ice packs or other more durable means to keep samples cool if they must be held for extended times or may be exposed to extreme heat. Use special insulated containers to avoid volatilization, particularly in warm climates. The temperature sensitivity of the coating should also be checked to determine if packing in ice may have a detrimental effect on the sample by causing solidification, separation, or any other change in its properties. Other samples which are not volatile at low temperatures, such as those with boiling points higher than 100°F, may be packed and shipped at room temperature. The MSDS on coatings and dilution solvents supply sufficient information to determine the coating properties.
3. When packaging samples for shipment, use bubble packing or crumpled paper to line the bottom of the box. The box should be firm, adhering to specifications given by the US Department of Transportation - DOT 12-B.^{8,9} Place the sample container in the box with the top up. Pack additional bubble pack or paper around the container so that the entire inner space of the box is sufficiently filled, to keep the sample container in the upright position and to prevent the container from shifting while in transit.
4. Enclose both the completed C of C form and a stamped envelope addressed to the agency for return of the C of C form in a larger envelope addressed to the analytical laboratory to which the sample is being sent. Place it on top of the packing over the sample containers and close the box.
5. Seal the box with strapping tape.

6. Paint and coating samples are considered hazardous material and should be shipped as a restricted article by Federal Express or another suitable carrier that can provide special handling. Label the outside of the box with sender/receiver addresses. If used, insert the completed air bill/cargo bill in its proper pocket and seal.
7. For all mail, courier, or special delivery service shipping, the box should have the following designations:
 - On top of box: "TOP - THIS SIDE UP."
 - On all sides of box: arrows pointing up.
 - On bottom of box: "OTHER END UP."
 - On one side of box: "Flammable liquid UN No. 1263."
 - On opposite sides of box: "Danger" and "Flammable Liquid" labels.
 - On fourth side of box: Name and address of the person shipping the samples.
 - On top of box: Shipping destination name and address along with any additional labels required by shipping company, such as the restricted article form used by Federal Express.
8. **DO NOT HOLD.** Ideally, a sample should be delivered to the laboratory for analysis on the same day it is obtained. If a sample must be stored overnight, it should be kept in a secure place, away from extreme temperatures and away from danger of breakage, leakage, or tampering, preferably in a locked storage cabinet. Maintain a sample log for those samples stored overnight. The log should contain the following information:
 - Sample number.
 - Facility name and location.
 - Date/time sample obtained.
 - Date sample was placed into locked storage cabinet.
 - Date when sample was mailed to the laboratory for analysis.

NOTE: SAMPLES SHOULD NOT BE MAILED ON A FRIDAY BECAUSE THEY WILL NOT BE DELIVERED UNTIL MONDAY. THIS MAY INTRODUCE SOME SUBSEQUENT QUESTION ABOUT ADEQUATE CHAIN OF CUSTODY OR SAMPLE INTEGRITY.

6. REFERENCES

1. Standard Practice for Sampling Liquid Paints and Related Pigmented Coatings, ASTM D3925-81 (Re-approved 1985). 1990 Annual Book of ASTM Standards, Volume 6.01, Philadelphia, PA, 1990.
2. Standard Methods for Sampling of Testing Shellac Varnish, ASTM D1650-76, 1990 Annual Book of ASTM Standards, Volume 6.02, Philadelphia, PA, 1990.
3. Standard Practice for Manual Sampling of Petroleum Products, ASTM Method D4057-88, Volume 5.01, Philadelphia, PA, 1990.
4. Standard Practice for Sampling Industrial Chemicals, ASTM Method E300-86, Volume 6.03, Philadelphia, PA, 1990
5. EPA-340/1-88-003, Recordkeeping Guidance Document for Surface Coating Operations and the Graphics Arts Industry, U.S. EPA, Stationary Source Compliance Division, Washington, DC, May 1989.
6. "Information on Sample Taking and Sample Transport/ Field Sampling Guidelines," (Draft), Bay Area Quality Management District, San Francisco, California, July 1990.
7. Code of Federal Regulations, Title 29, Part 1910, Occupational Safety and Health Standards, Department of Labor, July 1, 1989.
8. Dangerous Goods Regulations, 31st Edition, International Air Transport Association, Montreal, Quebec, Canada, 1990.
9. Code of Federal Regulations, Title 49, Part 178, Subpart F- Specifications for Fiberboard Boxes, Specification 12-B, Department of Transportation (49 CFR 178.205), October 1, 1989.
10. EPA-450/3-84-019, Procedures for Certifying Quantity of Volatile Organic Compounds Emitted by Paint, Ink, and Other Coatings, U.S. EPA, OAQPS, ESED, Research Triangle Park, NC, December 1984.
11. "EPA Region II Sampling Procedure," (Draft), New York, New York, June 1990.
12. "EPA Region I: VOC Sampling/Analysis," (Draft), McCusker, Boston, Massachusetts, June 7, 1990.
13. "Sampling Techniques & Test Methods," (Draft), California Air Resources Board, Sacramento, California, July 30, 1990.

14. Federal Test Method Standard No. 141c, Paint, Varnish, Lacquer, and Related Materials; Methods of Inspection, Sampling, and Testing. General Services Administration, January 24, 1986.
15. EPA-600/9-76-005, Quality Assurance Handbook for Air Pollution Measurement Systems: Volume I. Principles. U.S. EPA, EMSL, Research Triangle Park, NC, December 1984.
16. EPA-600/4-77-027b, Quality Assurance Handbook for Air Pollution Measurement Systems: Volume III. Stationary Source Specific Methods. U.S. EPA, EMSL, Research Triangle Park, NC, November 1976.

Note: References 10 through 16 were not directly cited in the Sampling SOP for RM 24/24A but the methods outlined in those references were generally incorporated. They are considered generally relevant and useful sources for sampling information.

**APPENDIX A
COATING DATA SHEET**

Date: _____

COATING DATA Source: _____

Data		
Coating: Supplier Name		
Name and Color of Coating		
Type of Coating (primer, clearcoat, etc.)		
Identification Number for Coating		
Coating Density (lbs/gal)		
Total Volatiles Content (wt%)		
VOC Content (wt%)		
Solids Content (vol%)		
Diluent Properties: Name		
Identification Number		
Diluent Solvent Density (lbs/gal)		
VOC Content (wt%)		
Water Content (wt%)		
Exempt Solvent Content (wt%)		
Diluent/Solvent Ratio (gal diluent solvent/gal coating)		

NOTE: If the solids content is not available from the manufacturer as a volume percent, it should be calculated. A copy of this calculation must be provided.

APPENDIX B
CHAIN OF CUSTODY (C of C) STRIP SEAL

SEALED	Date: _____	SEALED
	Initials: _____	

APPENDIX C
SAMPLE CONTAINER LABEL

Agency: _____ Inspector: _____
Sample ID#: _____ Date/Time: _____
Source ID#: _____
Coating Name/Type: _____
Plant Witness (Signature): _____

Circle analysis required: RM 24 RM 24A

Exempt Solvents: _____
Special Handling: _____

APPENDIX D
CHAIN OF CUSTODY FORM

CHAIN OF CUSTODY RECORD

Agency Inspector/Address:		Source Description:			
		Inspection Time/Date:			
Sample ID No.	Description of Sample				
Type of Analysis to be Performed/Remarks:					
Person Responsible for Sample (Inspector's Signature):				Time	Date
Relinquished By:	Received By:	Time	Date	Reason for Transfer:	
Relinquished By:	Received By:	Time	Date	Reason for Transfer:	
Relinquished By:	Received By:	Time	Date	Reason for Transfer:	
Relinquished By:	Received By:	Time	Date	Reason for Transfer:	

CHAIN OF CUSTODY RECORD

Agency Inspector/Address: John Smith USEPA-Region II Air Compliance Branch 26 Federal Plaza New York, NY 10278		Source Description: <i>Use a code name or number to avoid revealing source/coating information</i> Inspection Time/Date: 1500 hrs/May 7, 1991		
Sample ID No.	Description of Sample			
12345	One (1) 8 fl. oz. can of red enamel coating from coating machine 12B, station 3. <i>If it is known that the coating contains an exempt solvent, or is a known (or suspected) water-borne coating, indicate this fact also. If the sample is a multi-part coating, indicate this here and identify the other sample ID numbers that are part of the multi-part coating. Identify the mix ratios of each component and the set-up time of the formulation.</i>			
Type of Analysis to be Performed/Remarks: Method 24 or 24A				
Person Responsible for Sample (Inspector's Signature:) John Smith			Time 1500	Date May 7 1991
Relinquished By: John Smith	Received By: Joe Andrews	Time 1700	Date May 7 1991	Reason for Transfer: to custodian
Relinquished By: Joe Andrews	Received By: Bill Williams	Time 0900	Date May 8 1991	Reason for Transfer: to shipping dept.
Relinquished By: Bill Williams	Received By: -----	Time 1000	Date May 8 1991	Reason for Transfer: shipped Fed Ex to analytical lab
Relinquished By: -----	Received By: Mary Long	Time 1100	Date May 9 1991	Reason for Transfer: received at lab for analysis

--EXAMPLE--

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**APPENDIX E
DOT SPECIFICATION 12-B
FOR FIBERBOARD BOXES AS
SHIPPING CONTAINERS**

Subpart F—Specifications for Fiberboard Boxes, Drums, and Mailing Tubes

SOURCE: 29 FR 18951, Dec. 29, 1964, unless otherwise noted. Redesignated at 32 FR 5606, Apr. 5, 1967.

§ 178.205 Specification 12B; fiberboard boxes.

§ 178.205-1 Compliance.

- (a) Required in all details.
- (b) [Reserved]

§ 178.205-2 Definitions.

(a) Terms such as "200-pound test" mean minimum strength, Mullen or Cady test.

(b) "Joints" are where edges of parts of box, except recessed flanged heads, are connected together in setting up the box. Generally done by box maker.

(c) "Seams" are where edges of parts of box are visible, except joints, when box is closed.

§ 178.205-3 Classification of board.

(a) Fiberboard is hereby classified by strength¹ of completed board as in first column of the following table; weights specified in the table are the minimums authorized.

Classified strength ¹ of completed board	Solid fiberboard—Minimum combined weight of component piles exclusive of adhesives (pounds per 1,000 sq. ft.)	Facings for corrugated fiberboard	
		Double faced—Minimum combined weight of facings (pounds per 1,000 sq. ft.)	Double wall—Minimum combined weight of facings including center liner (pounds per 1,000 sq. ft.)
175 _____	149	75	
200 _____	190	84	92
275 _____	237	138	110
325 _____	237	138	110
350 _____	283	180	126
375 _____	283	180	180
400 _____	283	180	180
450 _____	283	180	180

¹ Mullen or Cady test (minimum).

(b) [Reserved]

¹ Mullen or Cady test (minimum).

§ 178.205-4 Solid fiberboard.

- (a) To be 3-ply or more; both outer plies water resistant.
- (b) [Reserved]

§ 178.205-5 Corrugated fiberboard.

- (a) Both outer facings water resistant; corrugated sheets must be at least 0.009 inch thick and weigh not less than 26 pounds per 1000 square feet; all parts must be securely glued together throughout all contact areas.
- (b) [Reserved]

§ 178.205-6 Stitching staples.

Stitching staples must be made in such a configuration that their holding capability as installed will not be less than that of flat steep staples $\frac{1}{8} \times 0.019$ inch in cross section and not less than $\frac{1}{8}$ -inch wide.

[Amdt. 178-12, 35 FR 11686, July 22, 1970]

§ 178.205-7 Tape.

(a) Coated with glue at least equal to No. 1½ Peter Cooper standard. Cloth tape of strength, across the woof, at least 70 units, Elmendorf test. Sisal tape of 2 sheets of No. 1 Kraft paper, total weight 80 pounds per ream (500 sheets, 24"×36"); sheets to be combined with asphalt and reinforced by unspun sisal fibers completely embedded in the asphalt and extending across the tape, except as provided in § 178.205-11(d). Other tapes of equal strength and efficiency are authorized.

(b) Tape for closure of slotted containers complying with the following requirements is authorized when applied as prescribed in § 178.205-17(a)(3):

(1) Tape must be not less than 3 inches wide and shall be made of two sheets of 100 percent sulfate Kraft each not less than 30 pounds basis weight, reinforced with glass, sisal, or rayon fiber, combined with a laminant of asphalt or other material not affected by temperature extremes any more than would standard 180°F. to 200°F. softening point asphalt.

(2) Tape must be reinforced by lengthwise fibers spaced not more than an average of $\frac{1}{2}$ inch apart, and by crosswise fibers spaced not less than an average of 2 per inch except that when a diamond pattern is em-

ployed for crosswise reinforcement, the spacing between the parallel sides of the diamond measured in the machine direction must be not more than 1 inch.

(3) Glass or sisal reinforced tape must have a minimum tensile strength in the machine direction of 75 pounds per inch of width and a minimum tensile strength in the cross direction of 45 pounds per inch of width; rayon reinforced tape must have a minimum tensile strength in the machine direction of 57 pounds per inch of width and a minimum tensile strength in the cross direction of 27 pounds per inch of width with elongation not exceeding 15 percent. Tensile tests on the finished product shall be made on a 3-inch width sample.

[29 FR 18951, Dec. 29, 1964. Redesignated at 32 FR 5606, Apr. 5, 1967, and amended by Amdt. 178-51, 43 FR 48645, Oct. 19, 1978]

§ 178.205-8 Test.

(a) Acceptable board must have prescribed strength, Mullen or Cady test, after exposure for at least 3 hours to normal atmospheric conditions (50 to 70 percent relative humidity), under test as follows:

(1) Clamp board firmly in machine and turn wheel thereof at constant speed of approximately 2 revolutions per second.

(2) Six punctures required, 3 from each side; all results but one must show prescribed strength.

(3) Board failing may be retested by making 24 punctures, 12 from each side; when all results but 4 show prescribed strength the board is acceptable.

(4) For corrugated fiberboard, doublepop tests may be disregarded.

(b) [Reserved]

§ 178.205-9 Types authorized.

(a) To be of solid or corrugated fiberboard of the following types, or as specifically provided for in § 178.205-19 to § 178.205-37:

(1) Slotted box; three-piece box without recessed ends; three-piece box of solid fiberboard with recessed ends; double-slide box; triple-slide box; telescope box, with sections of equal depth, or with covers, top or bottom or

both, with 3 inches overlap. (See § 178.205-14 (d) for boxes with single-flap closures.)

(2) [Reserved]

§ 178.205-10 Forming.

(a) Parts must be cut true to size and so creased and slotted as to fit closely into position without cracking, surface breaks, separation of parts outside of crease, or undue binding.

(b) [Reserved]

§ 178.205-11 Joints.

(a) For solid and corrugated fiberboard slotted containers: Lapped 1½ inches from center of scoreline except as in § 178.205-12; stitched at 2½ inch intervals and within 1 inch of each end of joint; body joint must be double-stitched (2 parallel stitches) at each end of joint over 18 inches long.

(b) For corrugated fiberboard slotted containers only: One butt joint taped (See § 178.205-7) is authorized; 3 inch tape required for boxes over 30 pounds authorized gross weight and 2 inch tape for others.

(c) For triple and double slide boxes: Joints of all slides must be taped (see § 178.205-7) for stitched; 3-inch tape required for boxes over 30 pounds authorized gross weight and 2-inch for others.

(d) For corrugated fiberboard only: One butt joint taped inside and outside with strips of one thickness of sulphate paper not less than 2 inches wide extending entire length of joint and firmly glued to box. For boxes not exceeding 65 pounds gross weight, outside strip of sulphate paper to be of basis weight not less than 60 pounds and inside strip of sulphate paper to be of basis weight not less than 40 pounds testing not less than 40 pounds. For boxes exceeding 65 pounds gross weight, outside and inside with strips of sulphate paper which must each be of basis weight not less than 90 pounds testing not less than 90 pounds. Basis weight of paper shown is for 500 sheets, 24×36 inches.

(1) For glued lap joint, the sides of box forming joint must lap not less than 1¼" and be firmly glued throughout entire area of contact with a glue or adhesive which cannot be dissolved

in water after the film application has dried.

§ 178.205-12 Flanged heads.

(a) Must have 4 flanges, at least 1" long above fillet, on each head. Recessed flanged heads not authorized for boxes of corrugated fiberboard.

(b) [Reserved]

§ 178.205-13 Seams which are to be stitched.

(a) Overlap, if any, required to be at least 1½ inches from center of scoreline except as in § 178.205-12.

(b) [Reserved]

[29 FR 18951, Dec. 29, 1964. Redesignated at 32 FR 5606, Apr. 5, 1967, and amended by Amdt. 178-64, 45 FR 81573, Dec. 11, 1980]

§ 178.205-14 Flap closures.

(a) Fill-in pieces, of the same type fiberboard as used in construction of the container, are required where it is necessary to prevent an opening between the inner flaps, unless otherwise provided by paragraphs (b) and (c) of this section or by Part 173 of this chapter.

(b) If to be closed by adhesive, each inner flap must cover at least one-third of face; inner flaps must butt or have full overlap, or fill-in pieces must be used, unless otherwise provided by Part 173 of this chapter, except that fill-in pieces are not required when outer flaps have full overlap. Outer flaps must butt or have full overlap.

(c) In lieu of fill-in pieces between inner flaps which do not butt, the following is authorized when linings are not prescribed in § 178.205.16.

(1) Top and bottom pads the same dimensions as interior of container of solid or corrugated fiberboard at least 125 pound test (Mullen or Cady).

(2) Minimum combined weight of facings for corrugated fiberboard pads must be at least 52 pounds per thousand square feet.

(3) Minimum combined weight of component plies for solid fiberboard pads must be at least 114 pounds per thousand square feet, exclusive of adhesives.

(4) Complete inner box or boxes.

(d) Single-flap closures are authorized for boxes with one dimension not

over 2"; each flap must be scored and form one of the small faces of the box and lap at least 5" on one of the largest faces.

[29 FR 18951, Dec. 29, 1964, as amended by Order 67, 30 FR 7425, June 5, 1965. Redesignated at 32 FR 5606, Apr. 5, 1967]

§ 178.205-15 Linings (when prescribed by § 178.205-16).

(a) Of 1-piece to extend around 4 faces with joint at center of 1 face and with 4 flanges, at least 1½" long, on each end (corners may be mitered) to bend over the other 2 faces; also 2 pads to cover the other 2 faces. Pads may be omitted if closing flaps afford

3 thicknesses throughout face. For boxes with 1 dimension not over 3", one of the widest flanges may be lengthened to cover entire face and lap 6" on the adjoining face and the other flanges and the pads may then be omitted.

(b) [Reserved]

[29 FR 18951, Dec. 29, 1964, as amended by Order 67, 30 FR 7425, June 5, 1965. Redesignated at 32 FR 5606, Apr. 5, 1967]

§ 178.205-16 Authorized gross weight and parts required.

(a) The authorized gross weight (when packed) and the parts required are as follows:

Authorized gross weight (pounds)	Strength of fiberboard (minimum) Mullen or Cady test						
	Solid board			Doublefaced corrugated		Doublewall corrugated	
	Box	Lining ^a	Heads ^a	Box	Lining ^a	Box	Lining ^a
15.....	175		(^a)	175		200	
30.....	200		275	200		200	
40.....	275		350	275		200	
				200	175		
55.....	325		(^a)	325		275	
65 ^a	375		(^a)	375			
				275	175	275	
	275	175	350	200	200		

¹ For recessed heads when used. In other cases same as for the box.

² As prescribed in § 178.205-15. A complete box is acceptable in place of the lining.

³ Recessed heads are not authorized.

⁴ Except as otherwise authorized herein or by Part 173 of this chapter.

(b) Triple slide boxes authorized for gross weights as follows: Of board at least 175-pound test for 40 pounds; of board at least 200-pound test for 65 pounds.

[29 FR 18951, Dec. 29, 1964. Redesignated at 32 FR 5606, Apr. 5, 1967, and amended by Amdt. 178-85, 51 FR 5976, Feb. 18, 1986]

§ 178.205-17 Closing for shipment.

(a) Slotted container, by coating with adhesive the entire contact surfaces of closing flaps and fill-in pieces where required or as prescribed in paragraph (a) (1), (2), or (3) of this section.

(1) By stitching with staples as prescribed by § 178.205-6 at 2½-inch intervals along all seams (one 5-inch space allowed when necessary to permit use of stitching device); or with staples made of flat wire of hardness not less than equivalent of Rockwell B90, and

not less than 0.037 inch thick and not less than 0.074 inch wide, with not less than 1¼ inch crown, may be spaced not more than 5 inches apart. Such staples may be used across center seam where outside flaps meet in lieu of on both sides of center seam but need only be used where outside flaps overlay inner flaps; or staples made of arcuate wire of hardness not less than equivalent of Rockwell B90, and not less than 0.027 inch thick and not less than 0.095 inch wide, with not less than 1 inch crown, may be spaced not more than 5 inches apart. Such stitches when spaced not more than 2½ inches apart may be used across center seam where outside flaps meet in lieu of on both sides of center seam but need only be used where outside flaps overlay inner flaps.

(2) For fiberboard boxes containing not more than 1 inside metal can not

exceeding 1 gallon nominal capacity, and as otherwise authorized by Part 173 of this chapter, by application of 2 strips of pressure-sensitive tape not less than $\frac{1}{2}$ inch in width, 1 strip to be placed approximately equal distance over the seam of abutting outer flaps, the other at a right angle to the first and spaced approximately equal distance on the closure face; strips must be of sufficient length to extend not less than 1 inch beyond score lines on side and end panels. Tape shall have a minimum tensile strength of 160 pounds per inch of width; minimum adhesion value of 18 ounces per inch of width; and minimum elongation of 12 percent at break, or having a minimum longitudinal tensile strength of not less than 240 pounds per inch of width; minimum adhesion value of 18 ounces per inch of width and a minimum elongation of 3 percent at break.

(3) For slotted containers only, reinforced tape complying with the requirements of § 178.205-7(b) is authorized for application over the center seam only. Tape must extend over the ends of box not less than $2\frac{1}{2}$ inches.

(4) All closing flaps may be firmly glued with a hot-melt adhesive of 100 percent solid content of thermoplastic material which will maintain bond at temperature ranging from 20° F. below zero to 165° F. above zero. Adhesive must be applied in not less than eight stripes (except as specified below) on each inner flap, each stripe having a minimum width of $\frac{3}{16}$ -inch after compression. Stripes may not be more than $1\frac{1}{2}$ inches apart and not less than four stripes must be applied on each side of center seam on each inner flap for full length of flap overlap area with one stripe not more than $\frac{1}{2}$ -inch from each side of center seam. If less than eight such stripes are applied on each inner flap, adhesive must cover and securely bond not less than 25 percent of flap contact area with bonded areas extending to within $\frac{1}{2}$ -inch or less of center seam.

(5) For regular slotted containers, pressure-sensitive tape is authorized for application over the center seams only and extending not less than two inches over the ends of the box. Tape must be not less than 2 inches wide and have a plastic film backing of pol-

yester, polypropylene, or equivalent material. Tape must have a minimum tensile strength of 45 pounds per inch of width in the machine direction and not less than 55 pounds per inch of width in the cross direction and may not be affected by temperature extremes normally encountered during transportation. Boxes closed by means of this pressure-sensitive tape must be capable of passing performance tests prescribed in § 178.210-10.

(b) Double slide boxes or triple slide boxes, by coating the inner slides with adhesive, or by closing with reinforced tape capable of withstanding test prescribed by paragraph (b)(1) of this section; for single-flap closures as authorized for boxes with one dimension not over 2 inches, the flaps must be fastened to the body with adhesive.

(1) Boxes selected at random, containing dummy contents similar to that to be shipped and packed to authorized gross weight, closed with reinforced tape across the ends and onto opposite side panels at least 2 inches, must be capable of withstanding a drop on each end from a height of 4 feet onto solid concrete without closure failure.

(c) Fiberboard boxes with covers extending over sides but not to bottom, covers resting on walls of box, or telescope boxes of equal depth section, covers extending to bottom, must be secured by one of the following methods:

(1) By not less than three metal straps, one lengthwise and others at right angles thereto.

(2) When cover extends not less than 3 inches over the walls of the box, by coating with adhesive the entire contact area of the cover.

(3) Telescope boxes having equal depth sections may be closed by application of reinforced water activated tape or pressure sensitive tape under conditions and for commodities as prescribed in Part 173 of this chapter.

(d) When metal straps are specified, boxes must be strapped with the required number; size at least $\frac{5}{8}$ inch \times 0.015 inch.

[29 FR 18951, Dec. 29, 1964. Redesignated at 32 FR 5606, Apr. 5, 1967, and amended by Amdt. 178-41, 42 FR 28135, June 2, 1977]

§ 178.205-18 Marking.

(a) *On each container.* Symbol in rectangle as follows:

DOT-12B***

(1) Stars to be replaced by authorized gross weight (for example, DOT-12B40, etc.).

(2) Name and address or symbol of person making the mark specified in paragraph (a)(1) of this section and located just above or below that mark. Symbol, if used, must be registered with the Director, OHMT.

(3) When metal straps are prescribed, boxes must be marked "_____ (number) METAL STRAPS REQUIRED" just above or below the mark specified in this paragraph.

(4) Size of markings: Specification markings prescribed in paragraph (a)(1) of this section must be at least ½ inch high; other markings must be legible.

(b) [Reserved]

[29 FR 18951, Dec. 29, 1964. Redesignated at 32 FR 5606, Apr. 5, 1967, and amended by Amdt. 178-40, 41 FR 38182, Sept. 9, 1976]

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO. EPA-340/1-91-010		3. RECIPIENT'S ACCESSION NO.	
4. TITLE AND SUBTITLE Standard Procedure for Collection of Coating and Ink Samples for VOC Content Analysis by Reference Method 24 and 24A		5. REPORT DATE May 1991	
7. AUTHOR(S) Bruce A. Olson, Melinda K. Wood, John T. Chehaske		8. PERFORMING ORGANIZATION REPORT NO. 91-133-T4/S	
9. PERFORMING ORGANIZATION NAME AND ADDRESS Pacific Environmental Services, Inc. (PES) 560 Herndon Parkway, Suite 200 Herndon, Virginia 22070-5225		10. PROGRAM ELEMENT NO. WA 91-133	
		11. CONTRACT/GRANT NO. 68-02-4464	
12. SPONSORING AGENCY NAME AND ADDRESS U.S. Environmental Protection Agency Office of Air Quality Planning and Standards Stationary Source Compliance Division Washington, D.C. 20460		13. TYPE OF REPORT AND PERIOD COVERED Final	
		14. SPONSORING AGENCY CODE	
15. SUPPLEMENTARY NOTES SSCD, Organic Chemicals Section, 401 M St., S.W., (EN-34W) Washington, D.C. 20460 Phone: (703) 308-8668			
16. ABSTRACT <p>This standard operating procedure (SOP) document is prepared with the intent of providing simple step-by-step instructions, covering all aspects of sampling coatings and inks, for use by EPA, State, and local regulatory agencies nationwide. The instructions are presented in general terms as much as possible while providing sufficient details for actual field sampling. The procedure should be used in conjunction with existing health and safety programs and in accordance with existing EPA or other agency training guidelines.</p> <p>Standard procedures are presented for sampling and handling of coatings and inks that require analysis by EPA Reference Methods 24 or 24A (RM 24/24A) as found in 40 CFR 60, Appendix A. This SOP has been expanded from an original version obtained from EPA Region II. For the purpose of developing consistency among agency inspectors nationwide in terms of sampling conducted, it delineates the activities deemed proper and necessary to ensure that the sample taken is representative of the coating or ink as applied. If the activities are conducted as described, questions concerning the credibility of sampling performed can be avoided.</p>			
17. KEY WORDS AND DOCUMENT ANALYSIS			
A. DESCRIPTORS		B. IDENTIFIERS/OPEN ENDED TERMS	C. COSATI Field/Group
Air Pollution Coatings Inks Analysis Solvents		Sampling VOC ASTM Methods	Sampling of Coatings and Inks
18. DISTRIBUTION STATEMENT		19. SECURITY CLASS (This Report) Unclassified	21. NO. OF PAGES 36
		20. SECURITY CLASS (This page) Unclassified	22. PRICE